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REPORT SM-48461-Q4

PROGRAM TO DEVELOP AN INORGANIC SEPARATOR FOR A HIGH TEMPERATURE SILVER-ZINC BATTERY

PERIOD ENDING 29 JULY 1966

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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QUARTERLY PROGRESS REPORT

PROGRAM TO DEVELOP AN INORGANIC SEPARATOR FOR A HIGH TEMPERATURE SILVER-ZINC BATTERY

by

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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOREWORD

The research, development and testing activities discussed in this report were sponsored by Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio. This report covers the period April 29 through July 29, 1966, and was prepared under Project Work Order Number 177091-01.

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1.0 INTRODUCTION AND SUMMARY

This report provides a complete summary of the work performed under Contract NAS 3-7639 during the quarter ending July 29, 1966. All of the work planned for Task I and Task II has been completed except for certain multiple cell tests which were rescheduled in accordance with Technical Directive No. 1, dated April 19, 1966[1]. The additional work outlined in Technical Directive No. 1 has been completed and the results are reported in this document.

A 5 Ah cell has been designed utilizing a frame concept to compartmentalize the electrodes. This design has 5 silver and 4 zinc electrodes and has proved adequate for the cells being investigated in this program.

Long cycle life capability of the cell components has been demonstrated and as many as 3123 cycles have been achieved. Full 5 Ah multiple plate cells have been cycled for as many as 1465 cycles at 25°C. Multiple plate 5 Ah cells have also been cycled at 100°C for more than 140 cycles in fulfillment of contract requirements. Additional work is necessary on frames and frame sealants and zinc electrodes in order to fully realize the potential of this design approach.

Satisfactory operation of test cells in various orientations has been achieved and no significant difference in cell performance has been observed in cells operating on their side or inverted as compared to operation in the normal upright position. Selection of the best top closure material will be made upon completion of these tests. Thus far, Armalon felt appears to be a satisfactory material for this purpose.

The work outlined in Technical Directive No. 1 has been completed satisfactorily. Test cells in which a rigid frame sealant was used declined in capacity after high temperature testing and did not have nominal OCV (1.86 v). In cells where a flexible sealant was used, there was substantially less change after heating to 135°C for 3 hours.

Although generally satisfactory for use in this program, additional studies will be made on molded polysulfone and PPO cases, covers and frame parts in order to select the best material for cell component use.

Based on the favorable results obtained thus far, the 5 Ah multiple plate cells specified at the end of Task II will now be fabricated and tested as described in the work statement.

An outline of the work planned for the next quarter is given in this report and the total number of engineering hours expended to date for personnel assigned to this program are reported.

2.0 TECHINICAL DISCUSSION

2.1 Task I - Design of a Multiplate 5 Ah Cell

The grooved-frame multiple plate cell design described in SM-48461-Q2[2] and SM-48461-Q3[3] has proved to be adequate for the cells being investigated in this program. Basically this cell design consists of a molded case and cover fitted with the terminals and filling hole. After installing the cell components the cover is sealed to the case, the electrolyte is added and the filler hole is closed with a Bunsen valve set at 10 psi. The cell components (electrodes and separators) are subassembled into frames to compartmentalize the individual electrodes. Slight modifications of groove dimensions and spacings were introduced in the frame E' type. The cell design selected consists of 4 negative electrodes and 5 positive electrodes.

2.2 Task II - Component Testing

2.2.1 Electrodes

No new work was initiated on electrodes during the reporting period. Three single-electrode cells were continued on cycle test, however.

Test Cell ESC-B-139 was removed from cycle testing after 3123 straight cumulative cycles. Analysis of the cell showed that failure was due to separator breakage caused by warpage of the plastic frames. The cell has been cycled for 622 cycles at 19% depth of discharge based on the original capacity Q_o followed by 2501 cycles at 8% Q_o.

Test ESC-B-202 completed 1620 cycles: 212 of these were cycles at 54% Q_o and the other 1408 cycles were at 11% Q_o. Failure was due to capacity loss of the zinc electrode.

Test Cell ESC-B-205 completed 2110 cycles (42 cycles at 60% Q_o followed by 2068 cycles at 10% Q_o). Cell failure was caused by leakage through the separator-frame sealant resulting in internal shorting.

These results confirm the long cycle life capability of the system. With further sealant improvement and development of improved zinc electrodes, even longer cycle life is anticipated.

2.2.2 Multiplate Cell Testing

2.2.2.1 Cycling Data

Table I shows test data obtained to date on multiple plate cells fabricated with machined frames. The design is indicated by D-series (D-1, D-2, etc---). The frame type is referred to as E, E', E'', E''', each of which designate slight modifications of groove dimensions and spacings.

The frame sealants used are indicated by their trade names — RTV, Shell Epon, Ethylene-Propylene (uncured liquid EP), Epibond, BR-89 and BR-92, (Epoxies supplied by The American Cyanamid Company for cementing polysulfone) and All-Bond.

Table I shows generally that these cells are capable of long cycle life at room temperature. Cell failures are caused by sealant failures, frame warpage or low zinc electrode capacity. Frame warpage caused at least one separator in the assembly to crack resulting in decline in cell capacity and termination of the test.

The best results, overall, were obtained with design D-10, using the E''' frame and the All-Bond cement. However, the problem of frame warpage still exists, especially at high temperature, where better results were obtained with a flexible cement such as RTV (see Section 2.2.2.3 Technical Directive No. 1).

It is significant that as many as 1465 cycles were obtained at 25°C as shown in Table I with multiplate cell MC-15 in these cycle tests. Cell performance remained essentially unchanged for over 1,000 cycles at which time a slight decline in cell performance was noted. After cell failure at 1465 cycles, the cell was disassembled and analyzed. Inspection clearly showed failure of the frame sealant RTV. It is expected that even longer cell life could have resulted if the frame sealant had not failed.

Four of the twelve multiplate cell cycle tests successfully completed over 400 cycles, and two of these four multiplate cells achieved over 1,000 cycles. Multiplate cell MC-25 completed 1,054

TABLE I

MULTIPLATE CELL (MC) CYCLING DATA

(25°C Except When Indicated By*)

Cell No.	Design				Discharge				No. of Cycles	Remarks
	Code	Frame	Cement	Original Capacity Q_o	Period (hrs)	Rate (A)	Current Density (mA/cm^2)	Depth of Discharge % Q_o		
13	D-1	E	RTV	4.9	A	1	7	10	205	L. C.
14	D-2A	E	Ep. 901	5.3	A	1	7	10	325	F. W.
15	D-3	E'	RTV	4.9	A	1	7	10	1465	S
16	D-2B	E	E. P	5.0	A	1	7	10	262	L. C.
17	D-2	E	RTV	4.5	C	1	7	100	19	F. W.
18	D-4	E'	RTV	5.8	A	1	7	9	167	L. C.
19	D-5	E'	Ep. 901	5.1	A	1	7	10	163	F. W.
20	D-4A	E'	Ep. 901	5.1	C	1	7	100	18	F. W.
22	D-7	E'	RTV	6.2	A	1	7	8	499	L. C.
23	D-7	E'	RTV	6.0	B	2	14	17	170	L. C.
24	D-5A	E'	RTV	4.8	A	1	7	10	326	F. W.
25	D-5A	E'	RTV	6.7	B	2	14	16	1054	S
26	D-7	E'	RTV	6.3	A	1	7	8	438	L. C.
27	D-7	E'	RTV	6.3	C	1	7	100	9	S
28	D-8	E''	RTV	6.0	B	2	14	17	78	F. W.
29	D-8	E''	RTV	5.8	B	2	14	17	82	S
30	D-7A	E'	BR-89	6.8	B	2	14	15	156	F. W.
31	D-8	E''	Epibond	7.1	A*	1	7	7	155	L. C.
32	D-8	E''	BR-89	6.9	A*	1	7	7	113	L. C.
33	D-8	E''	BR-92	6.6	A	1	7	8	154	F. W.
45	D-8	E''	All-Bond	5.2	A	1	7	10	265	F. W.
48	D-8	E''	All-Bond	6.4	A	1	7	8	512	F. W.
56	D-10	E'''	RTV	5.6	A*	1	7	9	163	F. W.
57	D-10	E'''	RTV	5.6	A*	1	7	9	167	F. W.
59	D-10	E'''	All-Bond	6.9	A	1	7	7	204	t

Key

*Run at 100°C

A = 1/2 x 1/2 hr (discharge-charge)

B = 1/2 x 1 hr (discharge-charge)

C = 24 hrs (discharge-charge)

FW = Frame Warpage

LC = Low Capacity

S = Sealant Failure

t = Test in Progress

cycles before the frame sealant failed. A typical discharge curve for this cell is shown in Figure 1. The rest of the multiplate cells failed at different cycling intervals before frame warpage, sealant failure, or zinc electrode degradation resulted in termination of the cycle tests. Typical cell performance is shown in charge-discharge curves of multiplate cells MC-22 and MC-26 after 335 cycles and 250 cycles in Figure 2 and Figure 3.

Four multiplate cells were tested at 100°C during the report period. Again, frame warpage caused at least one separator to crack in the frame assembly, resulting in the termination of two of the four tests after completing over 160 cycles. Of special interest were cells MC-56 and MC-57 which completed 163 and 167 cycles as shown in Table I. These cells were heat sterilized at 135° for three hours before being placed on cycle test. Discharge curves for these cells (shown in Section 2.2.2.3, Technical Directive No. 1) display a high discharge plateau of about 1.5 V. Two other cells tested at 100°C continued to cycle before loss of zinc electrode capacity caused cell failure. Figure 4 shows a high voltage plateau of multiplate cell MC-32 at 100°C.

The results confirm the capability of these cells to fulfill the contract requirements but also emphasize the need for design, sealant and zinc electrode modifications.

2.2.2.2 Test Cells Operating in Various Orientations

One of the design objectives of this program is a cell usable in any operating position. In order to achieve this desired versatility, it was necessary to develop a technique for closing the tops of the separator-electrode pack so the electrodes would be retained in place when the cell was placed in any position. The materials that were evaluated as top closures included Armalon felt, All-Bond epoxy, Nylon felt, RTV, potassium titanate fibers and plastisols. Table II shows the results obtained on multiple plate cells tested in three different orientations; upright, flat and inverted.

The experimental work using Armalon felt, All-Bond and Epibond is now complete. The remainder of the tests using Nylon felt, RTV, potassium titanate fibers and plastisols are in process and these results will be reported when the tests have been completed.

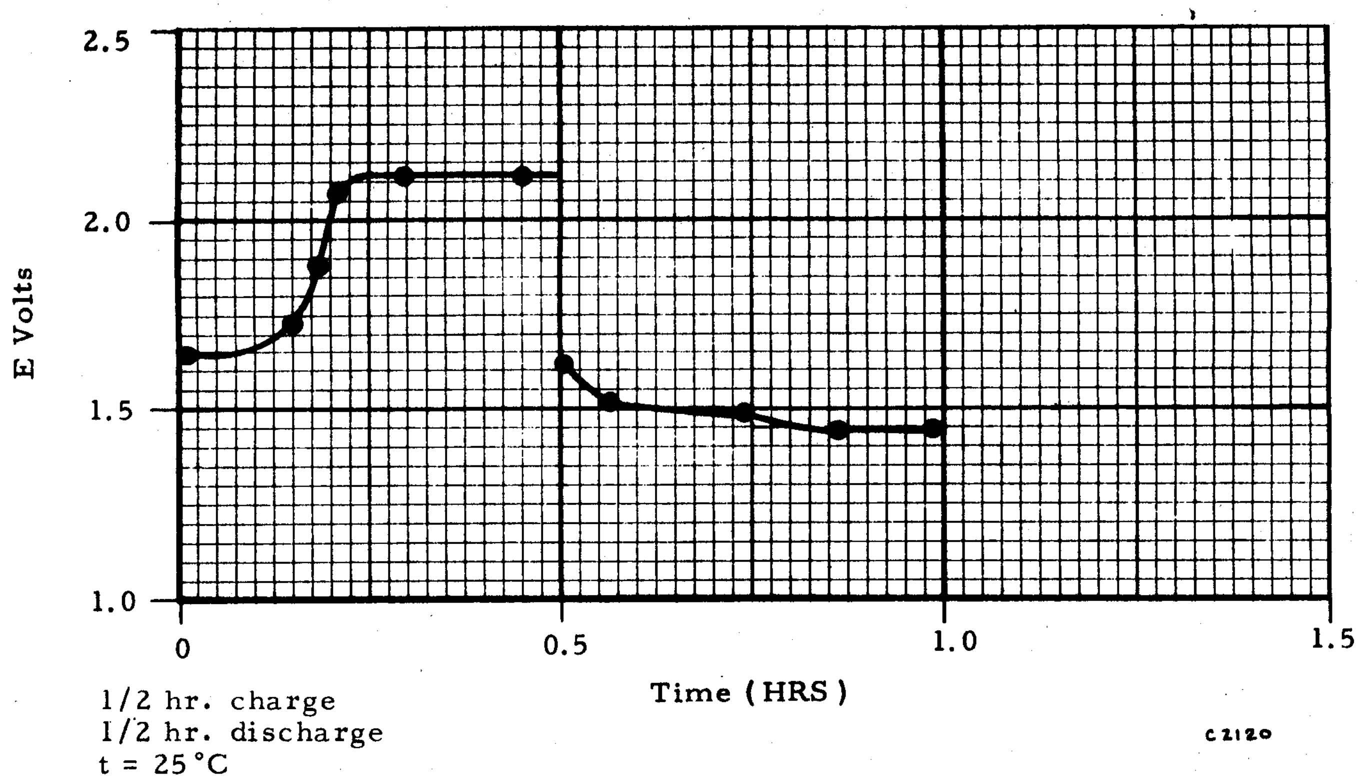


Figure 1. Test Cell MC-25 After 950 Cycles at $\frac{C}{5}$ at 25°C

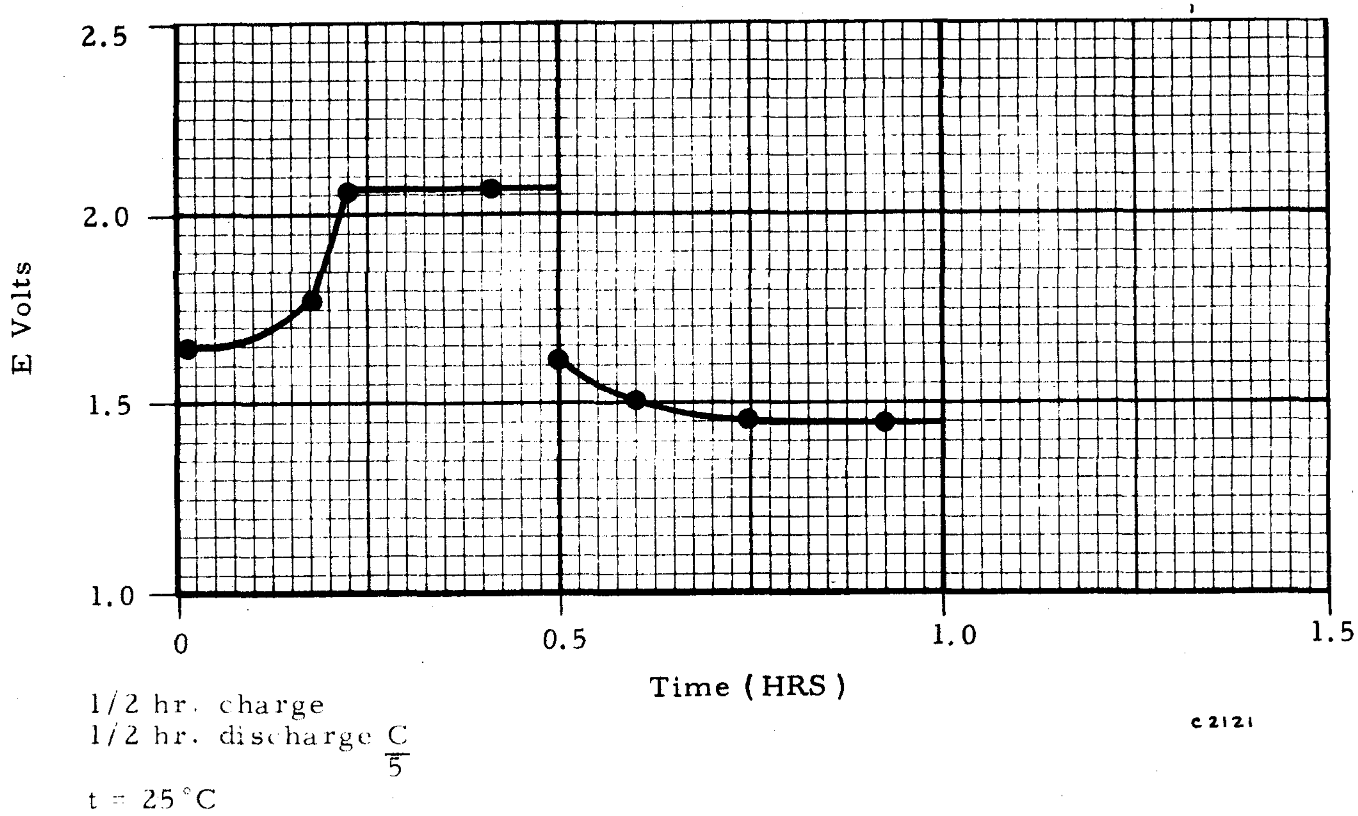


Figure 2. Test Cell MC-22 After 335 Cycles at $\frac{C}{5}$ at 25°C

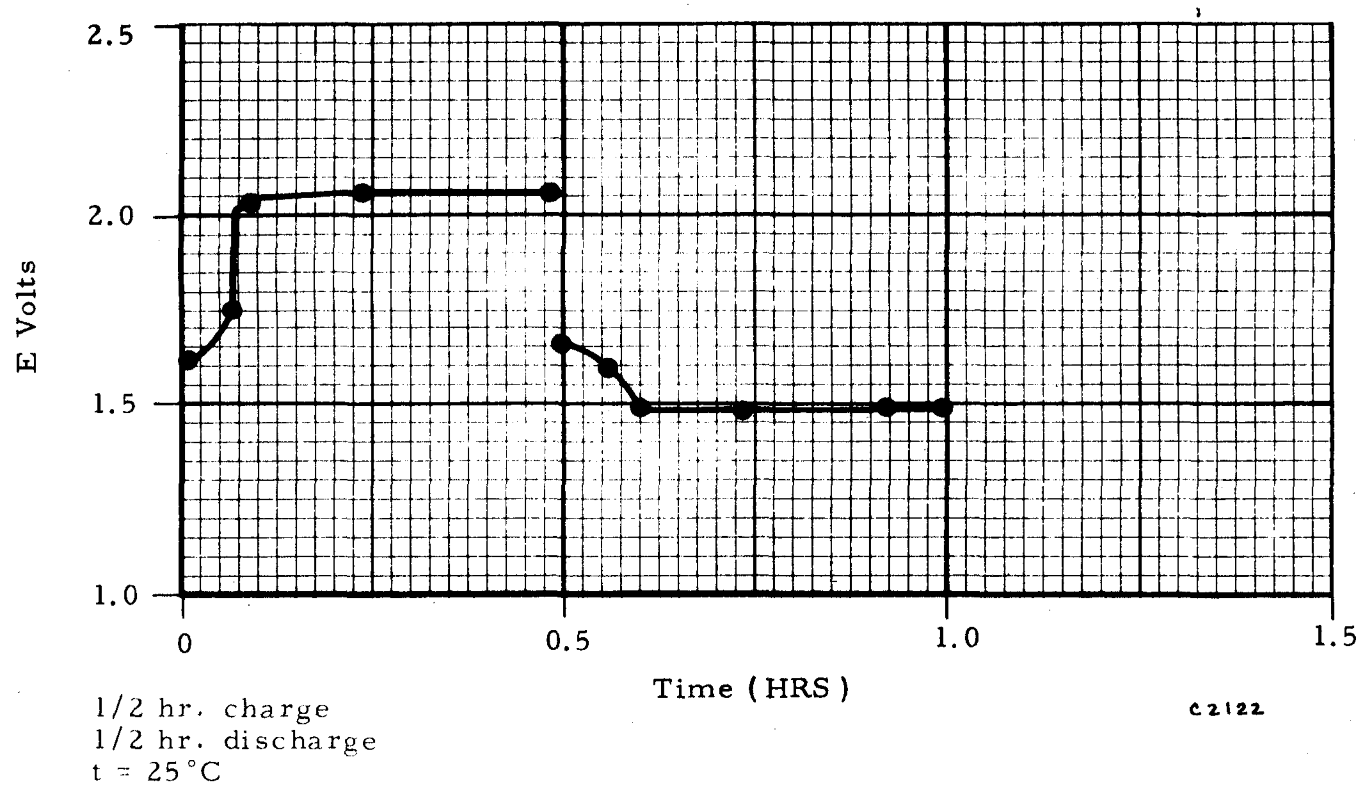


Figure 3. Test Cell MC-26 After 250 Cycles at $\frac{C}{5}$ at 25°C

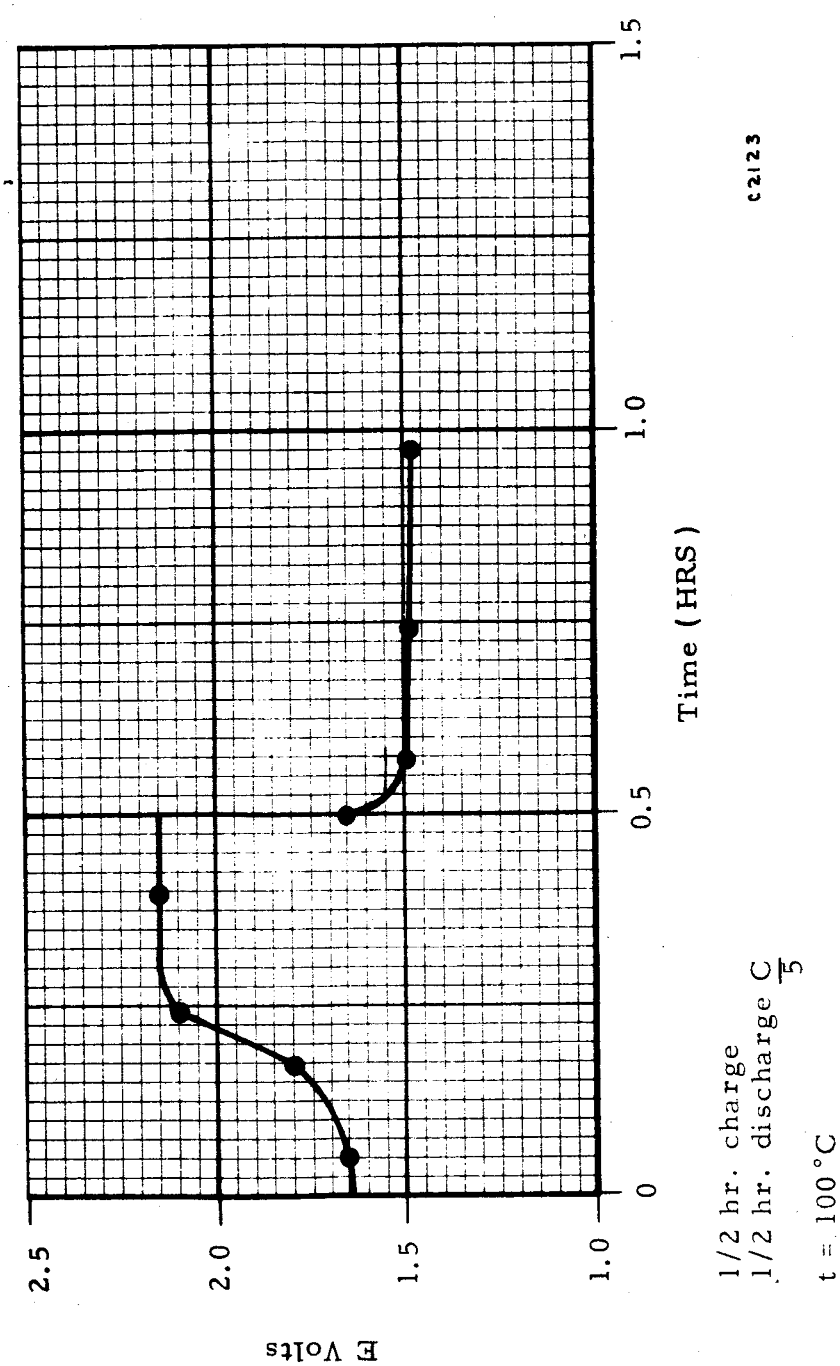


Figure 4. Test Cell MC-32 After 35 Cycles at $\frac{C}{5}$ at 100 °C

TABLE II

MULTIPLATE (MC) CELL CYCLING DATA AT 25°C AT DIFFERENT ATTITUDES

Cell No.	Design					Discharge					No. of Cycles
	Code	Frame	Cement	Original Capacity Q_o	Negative Compartment Top Filler	Period (hrs)	Rate (A)	Current Density	Depth of Discharge % Q_o	Mode	
42	D-8	E''	All-Bond	1.4	Armalon Felt	A	1	7	36	Up	39
43				5.2					10	Flat	276
44				5.0					10	Upside Down	259
45	D-8	E''	All-Bond	5.0	All-Bond	A	1	7	10	Up	265
46				5.3					10	Flat	515
47				5.1					10	Upside Down	339
48	D-8	E''	All-Bond	6.4	Epibond	A	1	7	8	Up	512
49				5.5					9	Flat	76
50				7.1					7	Upside Down	280
59	D-10	E'''	All-Bond	6.9	Nylon Fibres	A	1	7	7	Up	233 t
60				7.4					7	Flat	233 t
61				5.9					9	Upside Down	233 t
62	D-10	E'''	All-Bond	6.0	RTV	A	1	7		Up	229 t
63				6.6						Flat	229 t
64				4.4						Upside Down	229
65	D-10	E'''	All-Bond	5.7	U218X	A	1	7	9	Up	184 t
66				4.5					11	Flat	184 t
67				5.1					10	Upside Down	184 t

t = in test

Tests are continuing to establish a suitable material for closing the top of the electrode separator pack to enable cell operation at any attitude. Preliminary test data in Table II show cell cycle life from 259-512 cycles except cells MC-42 and MC-49. Cell MC-42 showed early signs of trouble attributed to cell fabrication.

Observation during cycling of the group with Armalon Felt in the negative compartments showed, however, that Armalon Felt does have a tendency to dislodge, especially if the cell is gassing appreciably. This was common at all cycling attitudes. Cells MC-43 and MC-44 cycled to 276 cycles before termination of the test
the Armalon Felt is a suitable top filler.

Cells MC-45, MC-46 and MC-47 used All-Bond epoxy resin in the negative while cells MC-48, MC-49 and MC-50 used another rigid epoxy, Epibond.

There was no apparent difference between these two cements with respect to cycle life. Multiplate cell MC-46 attained 515 cycles at 10% Q_o (All-Bond Cement) while MC-48 achieved 512 cycles at 8% Q_o (Epibond Cement). Figure 5 shows a typical discharge curve representing the cycling characteristics of the group using rigid cement top fillers.

Analysis of these cells after completing the cycle tests showed some zinc material lost through the small fill hole provided for electrolyte additions. All attitudes showed some zinc losses with most material being lost in the first mode. This is probably caused by the electrolyte pooling in the lower side of the cell.

Tests are now in progress using Nylon felt as a negative compartment top closure.

Multiplate cells MC-59, MC-60 and MC-61 have successfully completed 233 cycles and are continuing on test. No zinc loss has been observed to date.

Cell performance apparently is not affected by the cell orientation, i.e., upright, flat, or upside down. As can be seen

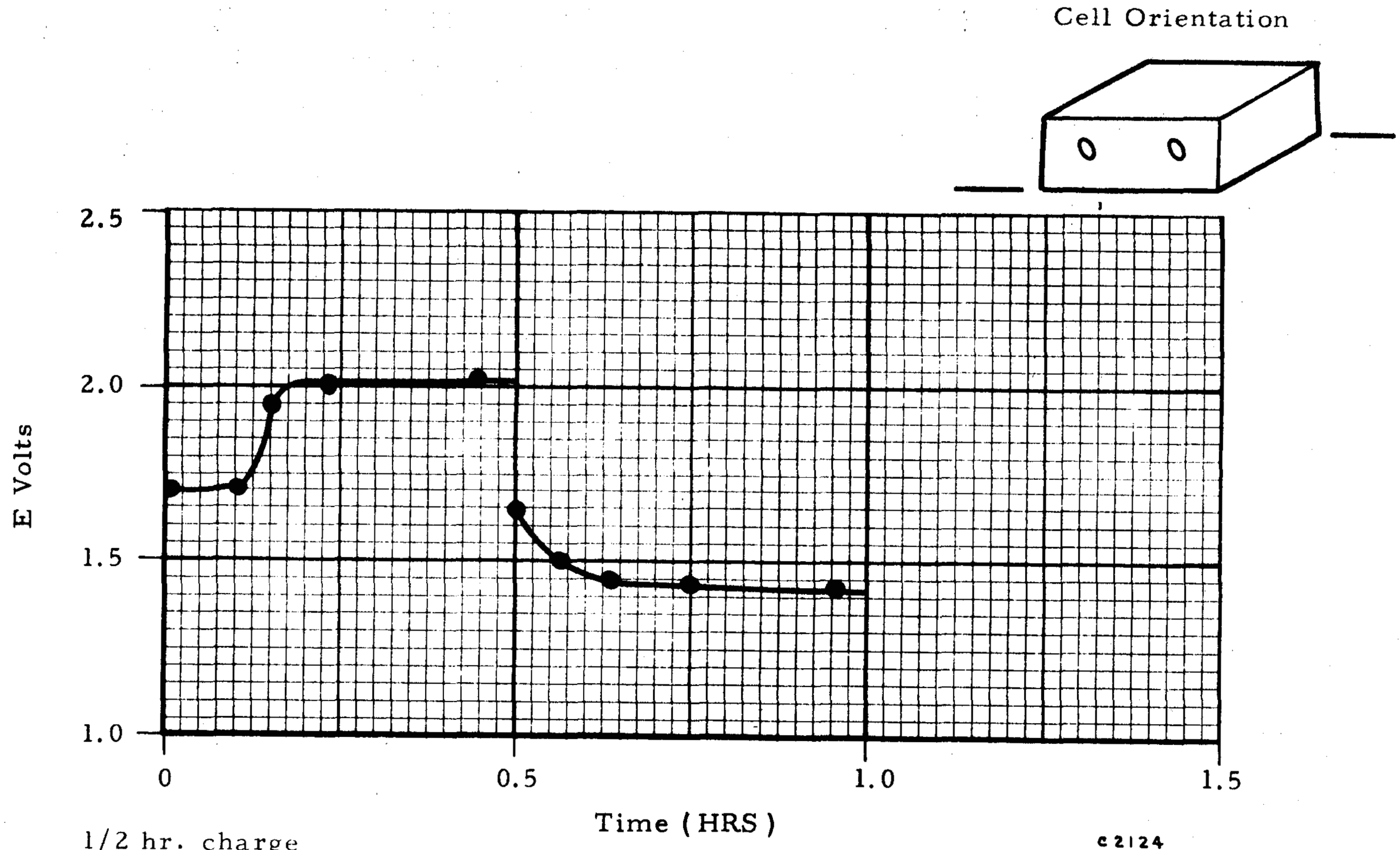


Figure 5. 5 Ah Cell MC-46 After 480 Cycles at $\frac{C}{5}$
Lying Flat On Side

from Figures 6, 7, and 8, discharge plateaus are similar at about 1.4 volts after 75 cycles at 10% of Q_o.

Two additional groups of cells are now being fabricated for attitude cycle tests. One group will use a flexible top filler (U-218X), while another group will use a flexible silicone rubber cement (RTV) as the top closure.

There appears to be no significant difference in cell performance cycling at any attitude. However, the most desirable negative compartment top closure appears to be a combination of a porous material such as nylon felt and a rigid epoxy cement used on top of the porous material leaving a small hole for adding electrolyte. This would provide an escape for gas, and insure that the porous material is held in place. This design is illustrated in Figure 9.

2.2.2.3 Technical Directive No. 1[1]

Technical Directive No. 1 was issued on April 19, 1966 in order to permit a preliminary evaluation of design configuration and components prior to fabricating the eight multiplate cells specified in the work statement (TASK II, paragraph 7). A copy of TD-1 is included in Appendix A of this report, as well as our test procedures.

All of the tests specified in TD-1 have been completed and the results are shown in Table III.

Six multiplate cells, MC-38, 39, 54, 55, 56 and 57 were fabricated and tested in accordance with the specification outlined in Technical Directive No. 1, issued April 19, 1966. All cells passed the required electrolyte leak tests. They were then given one formation cycle and then discharged at 3 A at 25°C (20 mA/cm^2). They were then discharged at 3 A at 100°C.

The internal resistance of each cell was measured three times at different times while charging on the argentic plateau. All measurements were consistent and very close. The average values reported in Table III are accurate to within 0.005 ohm.

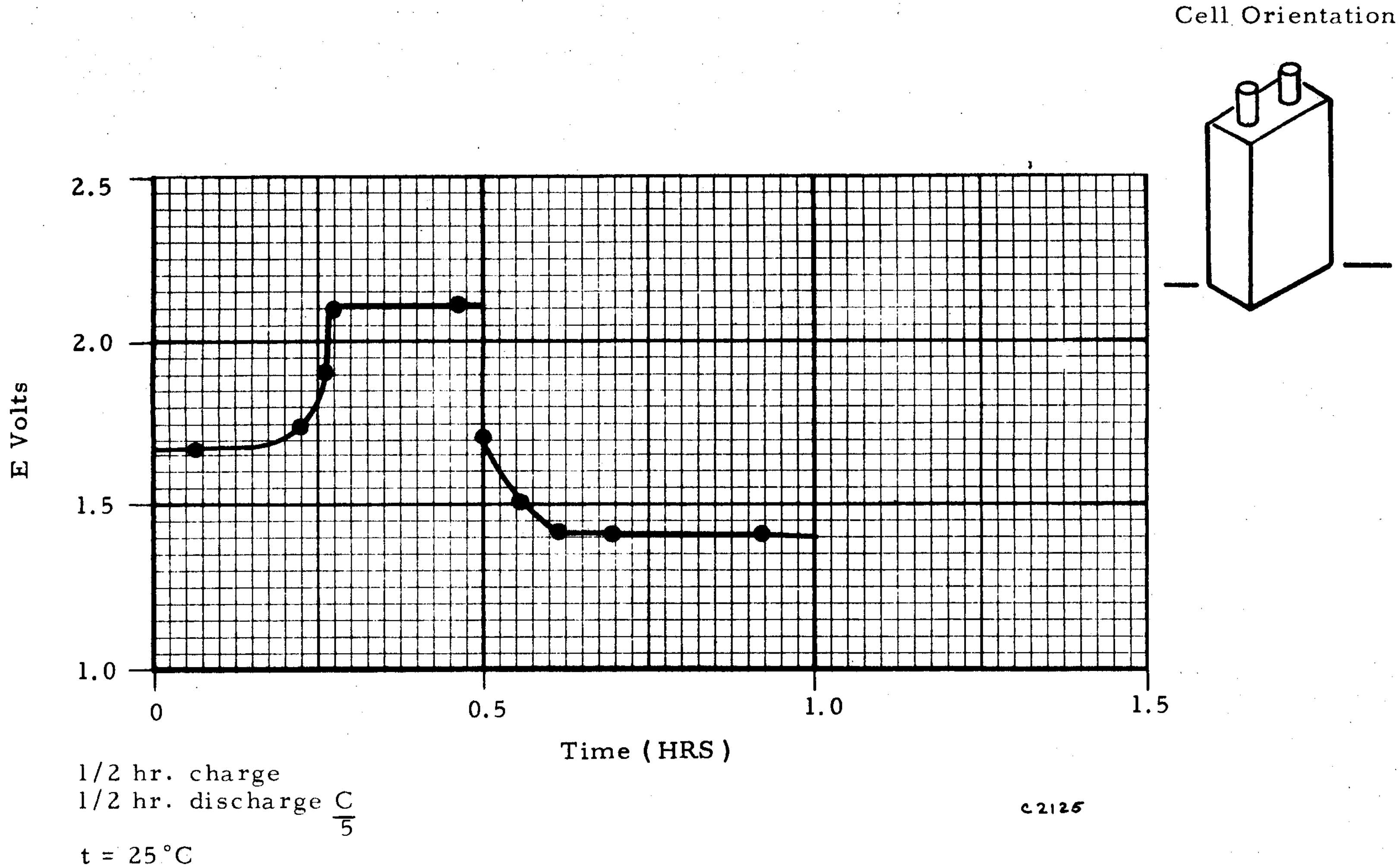


Figure 6. 5 Ah Cell MC-59 After 75 Cycles at $\frac{C}{5}$ in Upright Position

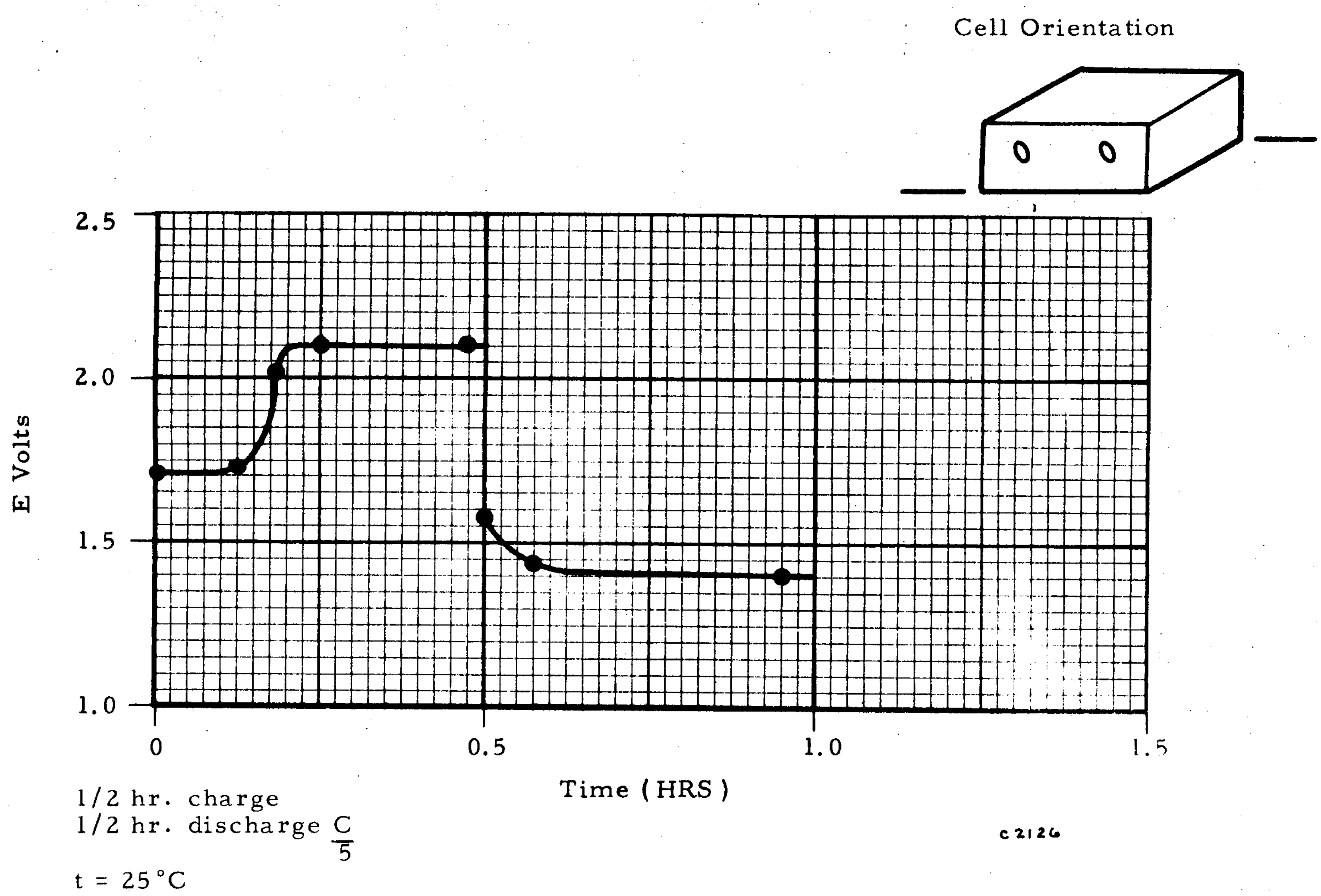
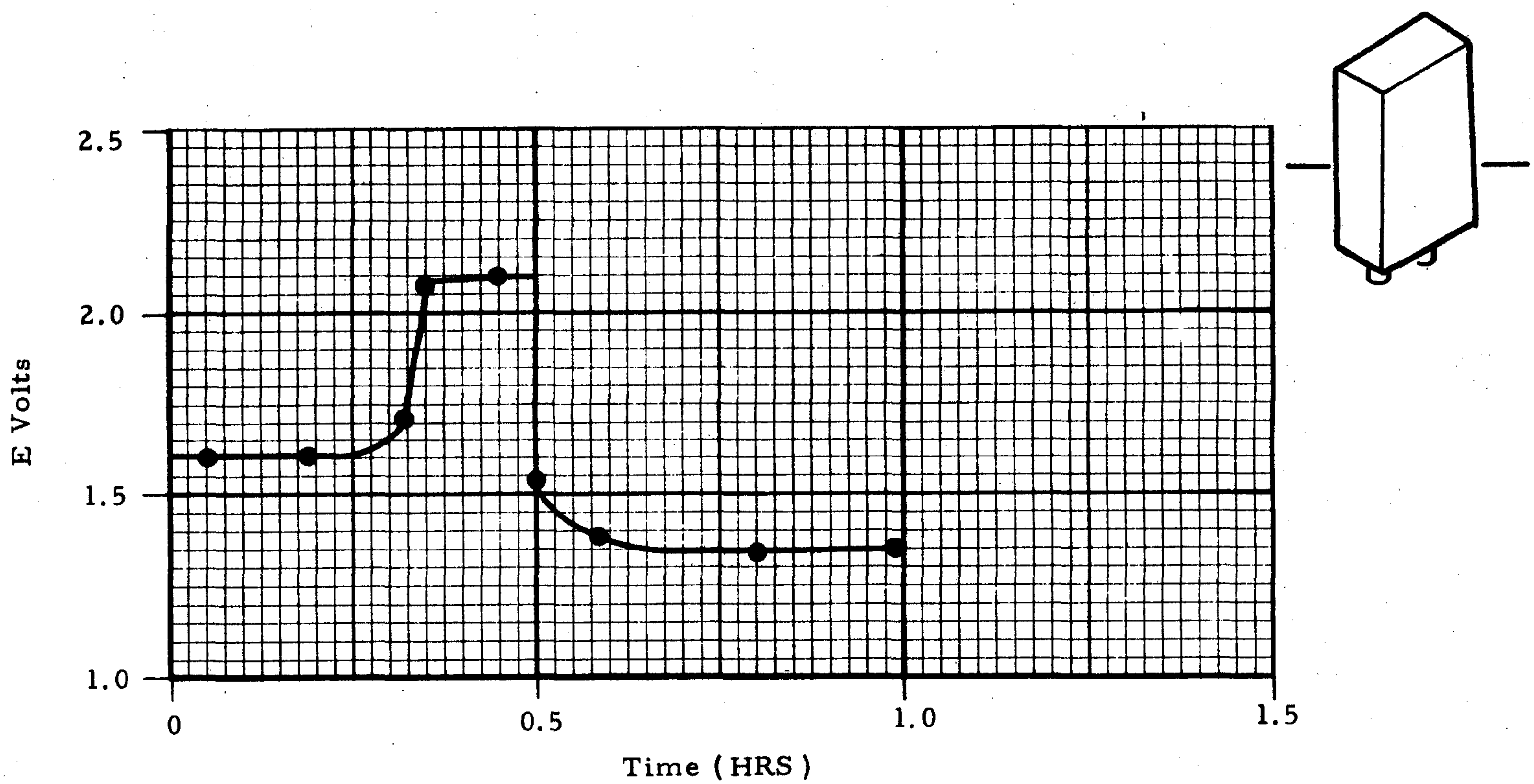


Figure 7. 5 Ah Cell MC-60 After 75 Cycles at $\frac{C}{5}$ Lying On Flat Side



1/2 hr. charge
1/2 hr. discharge $\frac{C}{5}$

t = 25 °C

Figure 8. 5 Ah Cell MC-61 After 75 Cycles at $\frac{C}{5}$ in Inverted Position

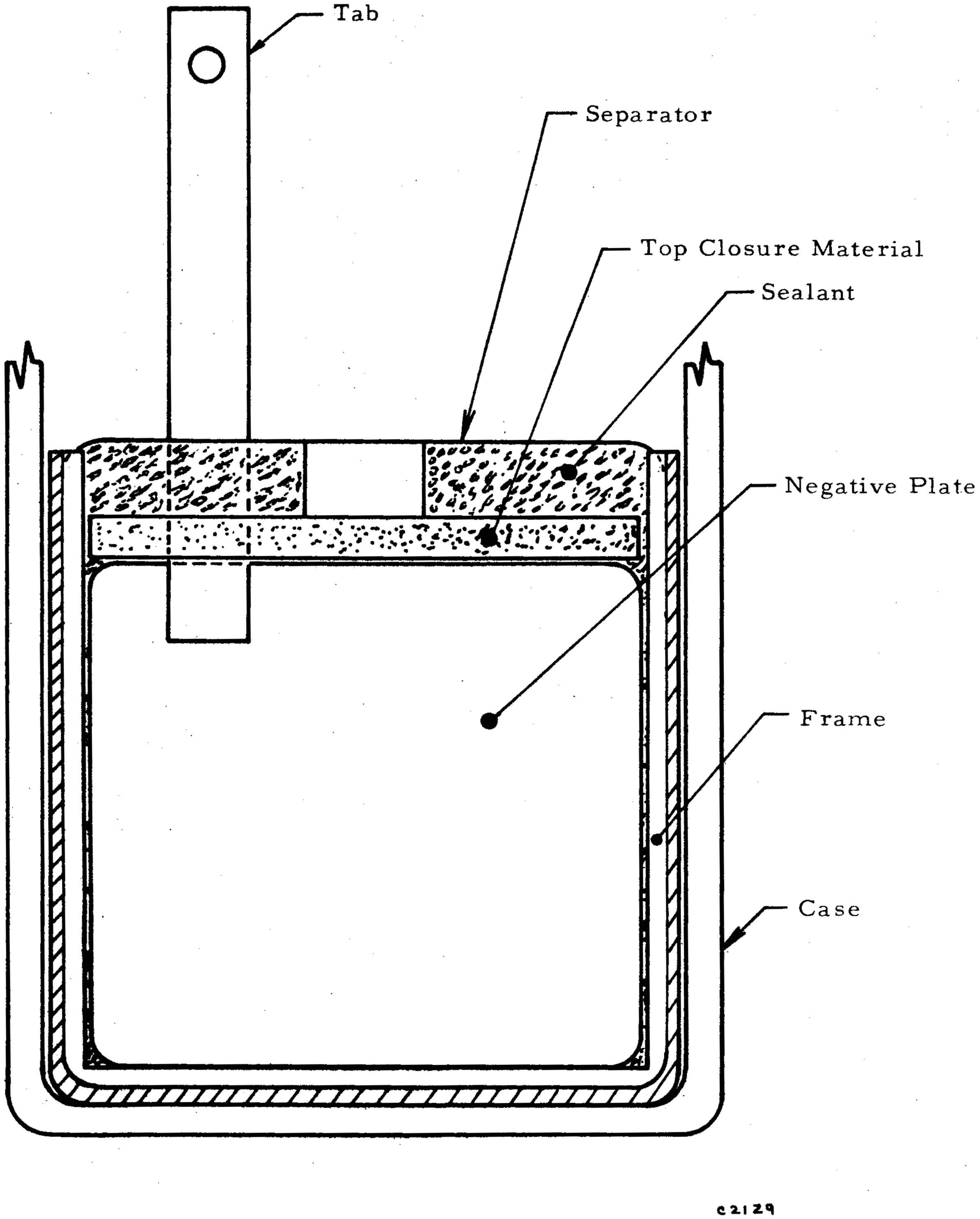


Figure 9. Top Closure For Negative Electrode Compartment

TABLE III

SUMMARY OF TEST RESULTS – TECHNICAL DIRECTIVE NO. 1

Cell No. and Cement	Outputs (Ah) at Cycles #1 1.5A 25°C	#2 3A 25°C	#3 3A 100°C	Internal Resistance (ohm.)	After Heat Exposure 3 hrs/135°C OCV	Capacity After Recharge
MC-38 All-Bond	5.5	4.8	4.2	0.057	1.58	1.2 Ah
MC-39 All-Bond	5.4	4.5	4.2	0.057	1.58	1.0
MC-54 All-Bond	5.1	5.4	6.0	0.063	1.76	1.2
MC-55 All-Bond	5.6	4.7	4.5	0.067	1.83	1.0
MC-56 RTV	5.6	5.7	6.3	0.06	1.86	2.0
MC-57 RTV	5.6	5.7	6.3	0.06	1.86	2.8

As can be seen from Table III the cells fabricated with epoxy cements (MC-38, MC-39, MC-54 and MC-55) lost their normal open-circuit voltage and exhibited a decline in capacity after the high temperature test.

Analysis of these cells showed separator fractures caused by plastic frame warpage. However, cells MC-56, MC-57 in which a flexible sealant (RTV) was used continued to maintain normal good open circuit voltage (1.86 V) and retained considerably greater capacity than the cells using the rigid cements.

Cells MC-56 and MC-57 were then placed on cycle test at 10% Q_o at 100°C and have successfully completed 160 cycles. Figures 10, 11, 12 show discharge curves for Cells MC-56 and MC-57 at the 5th and 140th cycles. It can be noted from these graphs that the discharge voltage plateaus are favorably high, averaging about 1.5 volts.

2.3 Inorganic Separator

Type 5-036-011 inorganic separators were prepared to meet program requirements during this quarter. Continuing process improvements, quality control tests and tighter inspection standards have resulted in improved uniformity and reproducibility. The high level of uniformity being regularly achieved is reflected in consistant cell performance.

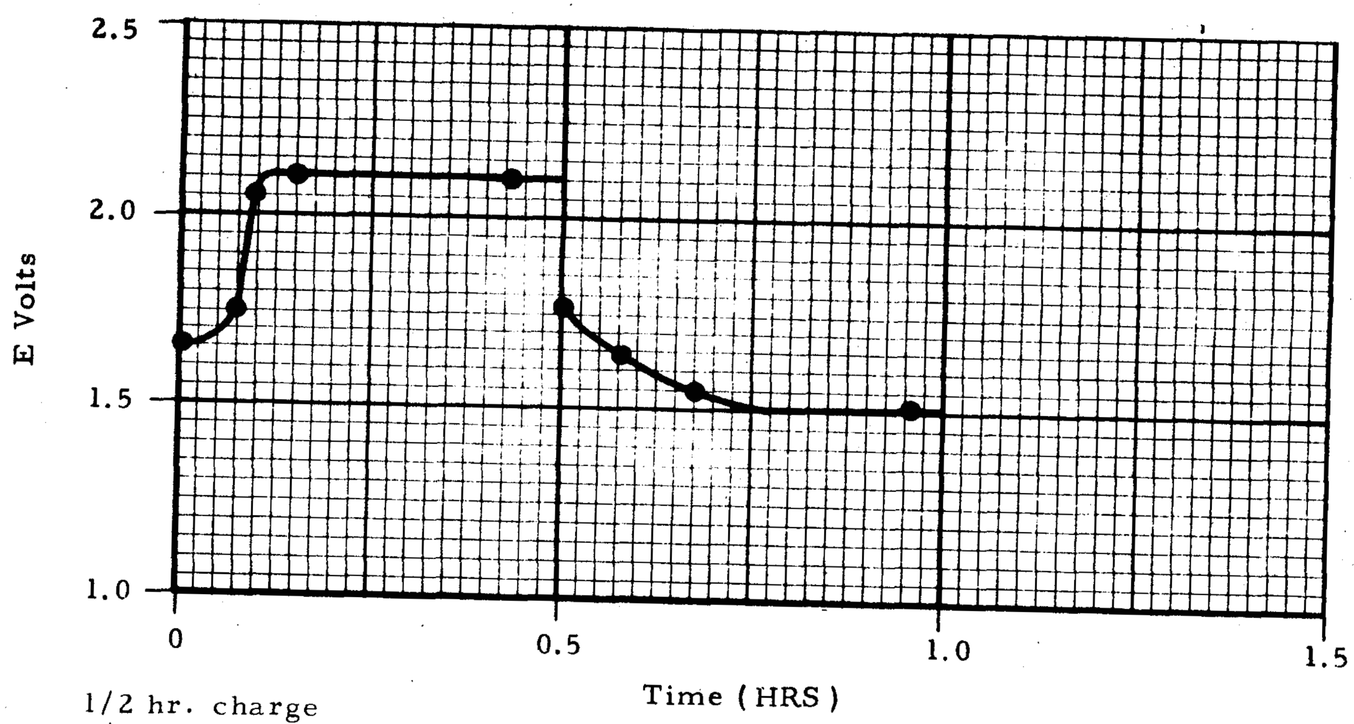
2.4 Cell Cases, Covers and Frames

2.4.1 Molded Cases and Covers

Molded polysulfone cases having 0.100 in. thick walls were obtained and evaluated. It was found that these cases satisfactorily passed leak testing for 10 minutes at 30 psig at 125°C. They were also leak tight at 150 psig at 25°C although bulging was observed in all tests. The test cases tested at 125°C retained a certain permanent bulge after cooling to room temperature. These data are shown in Table IV.

In these same tests the case to cover seals were also found to be satisfactory and showed no leakage up to 150 psig.

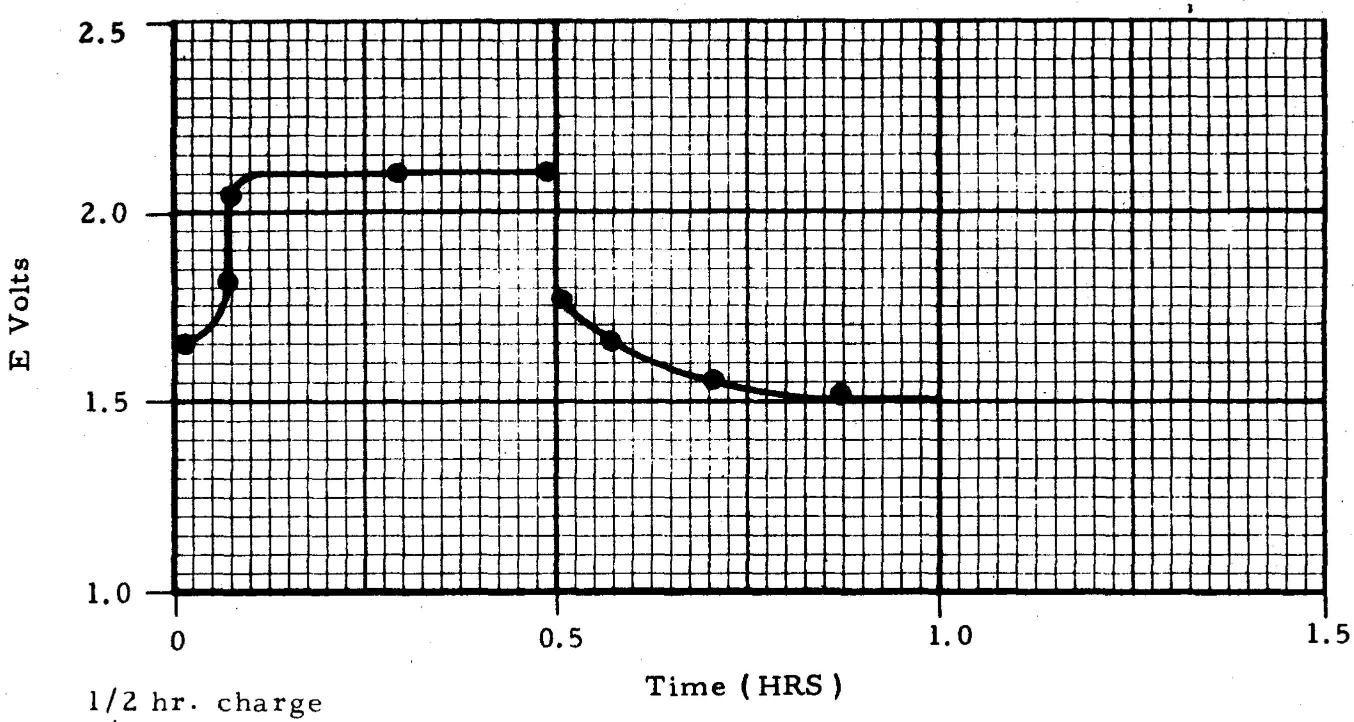
Although generally satisfactory for use in this program, prolonged heating of polysulfone cases in contact with KOH causes cracks to



1/2 hr. charge
1/2 hr. discharge $\frac{C}{5}$

t = 1

Figure 10. 5 Ah Cell MC-56 Fifth Cycle at 100 °C at $\frac{C}{5}$
After Heat Soak at 135 °C

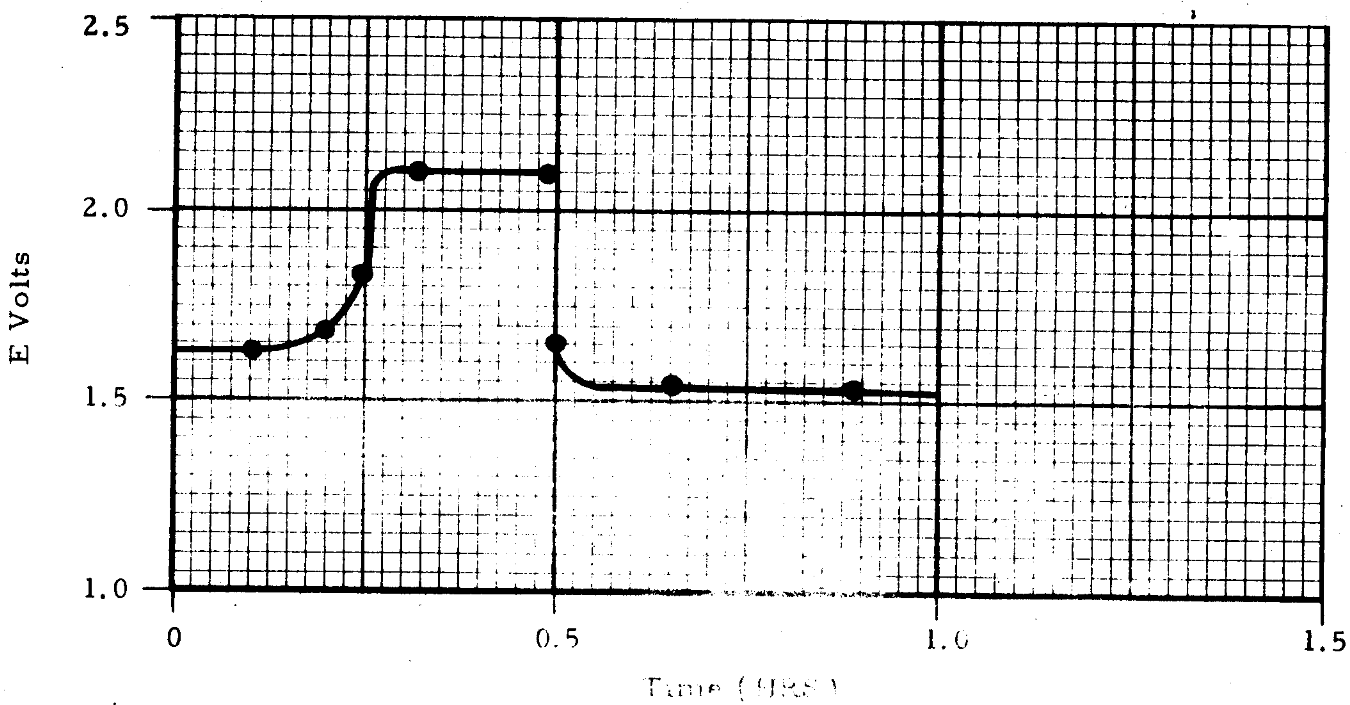


1/2 hr. charge
1/2 hr. discharge $\frac{C}{5}$

t = 100 °C

c2130

Figure 11. 5 Ah Cell MC-57 Fifth Cycle at 100 °C at $\frac{C}{5}$ After Heat Soak at 135 °C



1/2 hr. charge
1/2 hr. discharge $\frac{C}{5}$

t = 100 °C

C 2131

Figure 12. 5 Ah Cell MC-57 140 Cycles at 100 °C at $\frac{C}{5}$
After Heat Soak at 135 °C

TABLE IV

CASE PRESSURE TEST RESULTS

Material	Temp., °C	Pressure, (psig)	Time, (min)	Thickness, (in)	Increase in Thickness, (in)
PPO	25	0	0	0.996	0
	115	15	0	1.065	0.069
	125	30	0	1.125	0.129
	125	30	5	1.132	0.136
	25	0	5	1.102	0.006
	25	0	0	1.102	0
	125	30	0	1.135	0.033
	125	30	5	1.140	0.038
	125	30	10	1.142	0.040
	25	0	10	1.111	0.009

Permanent Bulge: First Test: 0.006"
Second Test: 0.009"

Material	Temp., °C	Pressure, (psig)	Time, (min)	Thickness, (in)	Increase in Thickness, (in)
Polysulfone					
(original)	25	0	0	1.000	0
	125	30	0	1.095	0.095
	125	30	5	1.101	0.101
	125	30	10	1.105	0.105
	125	30	15	1.110	0.110
(final)	25	0	15	1.002	0.002

Permanent Bulge: 0.002"

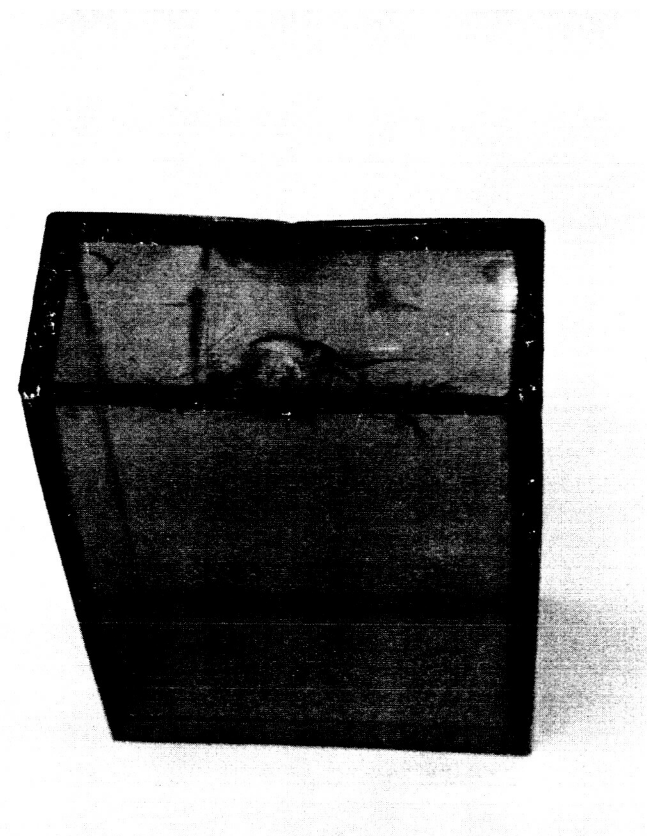
develop in this material. These cracks appear to be related to mold design and molding techniques rather than to the material itself. Additional work is being done on case molding and mold designs with the expectation that a substantial improvement in molded polysulfone cases will result.

In one test a molded polysulfone case was subjected to 111 hours at 145°C in 30% KOH to simulate thermal sterilization. As shown in Figure 13, small cracks developed in the bottom of the case as a result of this treatment. These cracks radiated outward from the sprue marks. Examination of molded cases that have not been heat treated using polarized light indicate this to be a high stress area and improved molding techniques should help to relieve this problem. Other than these cracks, the cases appeared to be unaffected by the sterilization cycle. Figure 14 shows the polarized light inspection method now being used for cover inspection.

It has been found that polysulfone is superior to polyphenylene oxide for molded cell case and component use. Cases molded from PPO were striated due to molding problems. It was determined that these striations represent weak places that cracked when the cases were filled with KOH and heated to 100°C. Cases molded from polysulfone are free from these striations and have proven to be much more satisfactory. Machined components made from polysulfone stock were also substantially more satisfactory than those machined from PPO which warps badly upon aging. Final selection of a case and cover material is being deferred until satisfactory PPO moldings are obtained so a fair evaluation of the two materials can be made. In the interim, all test cells will be fabricated with polysulfone cases.

2.4.2 Frames

Although polysulfone appears to be definitely superior to PPO as a frame material due to its greater dimensional stability on aging, frames machined from molded sheets exhibit a certain amount of warpage both at 25°C and 100°C. Molded polysulfone frames may be more dimensionally stable than machined frames. Distortion of the frames can cause leakage through the seal between the separator and the frame or even fracture the separator if the distortion is pronounced.



c1950

Figure 13. Cell Case Showing Cracks Radiating From Mold Marks

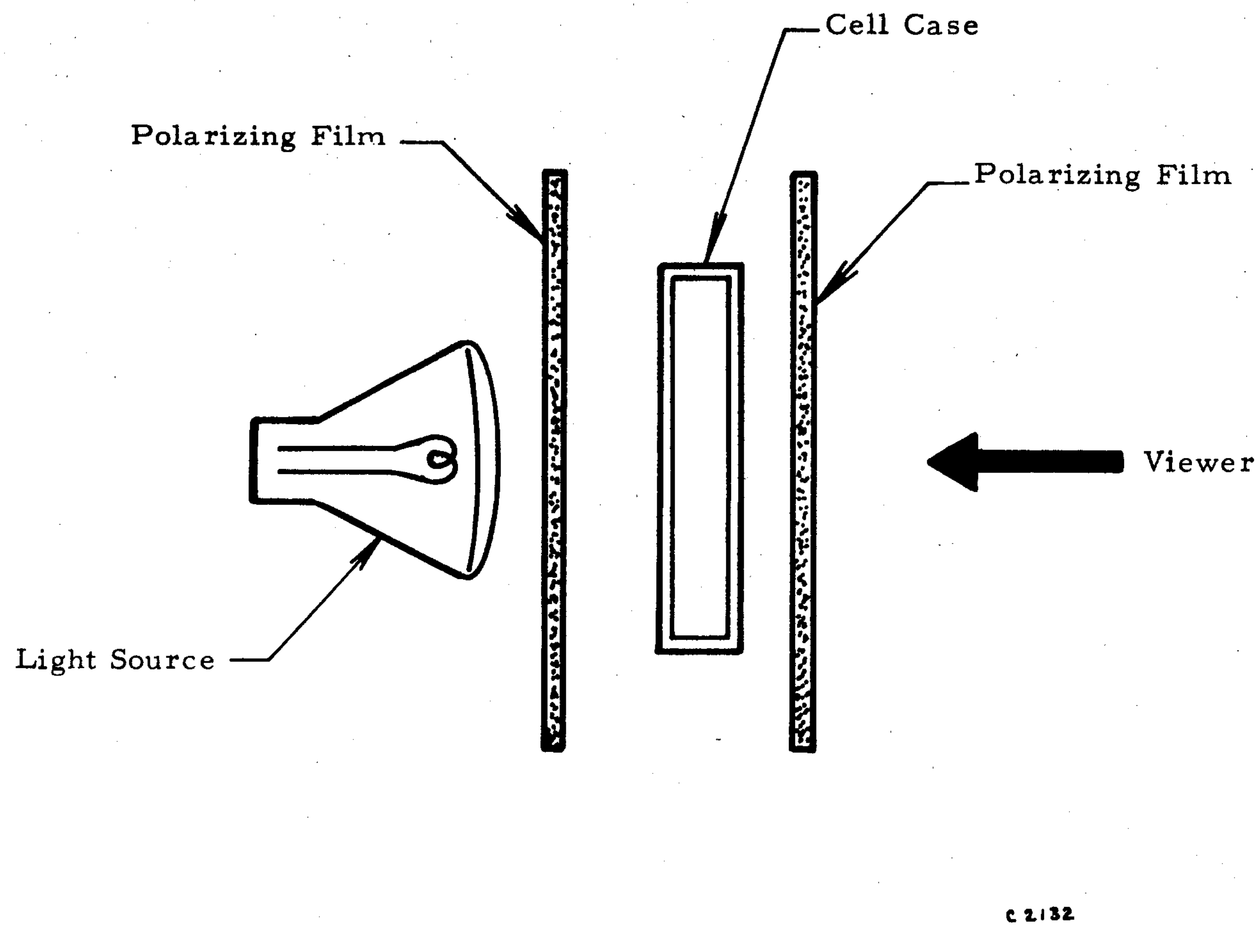


Figure 14. Polarized Light Inspection of Battery Component

The problem of sealing the separator satisfactorily into the frames is related to the sealant used,as well as to the frame material. The combination of frame, separator and sealant must be dimensionally stable, provide for a tight seal between compartments and be slightly flexible in order to permit the electrodes to expand and contract without breaking either the seals or the separator.

In recent tests excellent results have been obtained with assemblies in which the frames are molded in place around the separators and electrodes using slightly resilient materials such as All-Bond or plastisol sealants. These materials were used by either making a sub-assembly of the separators and zinc electrodes (wafers) or by making a sub-assembly of all cell components and then framing them with the sealing material. After proper curing, the subassembly is assembled in a multiplate cell case along with the remaining components in the usual manner. These configurations and tests results are discussed in detail in Section 2.2.2.1 of this report.

2.5 Results and Conclusions

The grooved-frame multiple plate cell design in which individual electrodes are compartmentalized by the inorganic separators have proved to be satisfactory for the 5 Ah cells being developed in this program. The selected cell design consists of four negative and five positive electrodes.

The long cycle life capability of this design approach is illustrated by the results obtained with test cells ESC-B-139, which completed 3123 cycles (622 at 19% and 2501 at 8% Q_o), test cell ESC-B-202 which completed 1620 cycles (212 at 54% and 1408 at 11% Q_o) and test cell ESC-B-205 which completed 2110 cycles (42 at 60% and 2068 at 10% Q_o on the 1/2-hour, 1-hour test regime. Because test cell failure is generally related to zinc electrode changes, frame distortion or failure of the frame sealant, improvement of the zinc electrode as a result of the work being done on contract NAS 3-8513 and further improvement of the frames and sealant will result in even longer cycle life.

In 5 Ah configurations 1465 cycles at 25°C have been successfully completed and three cells have exceeded 145 cycles at 100°C. These multiple plate 5 Ah cell tests confirm the capability of this system of meeting the contract requirements for this program but also emphasize the value of additional work on design, frame sealants and the zinc electrode in order to determine the full potential of this approach.

Investigation of cell pack top closure materials and methods have been initiated using a variety of materials such as Armalon felt, epoxies, nylon fiber, plastisols and silicone rubber. Satisfactory operation for as many as 515 cycles in all cell orientations has been obtained thus far. These tests are continuing in order to select the best material and techniques. As no significant differences in cell performance characteristics have been noted as a result of cell orientation, successful achievement of this design goal can be anticipated.

The work required by Technical Directive No. 1[1] has been completed. Test cells fabricated with rigid frame sealants exhibited a loss of capacity after high temperature testing, however, those assembled with flexible sealants retained capacity and exhibited nominal (1.86V) open circuit voltage. Test Cells MC-56 and MC-57 have completed 163 and 167 cycles at 10% Q_o.

Continuing improvements in inorganic separator technology, quality control and tighter inspection standards have resulted in higher levels of uniformity and reproducibility which are reflected in consistent cell performance.

Molded polysulfone cell covers with 0.100 in. thick walls were leak tight at 30 psig at 125°C and also did not leak when tested at 150 psig at 25°. Some bulging was noted. The case to cover seals were leak tight at 150 psig.

Additional work will be done on case molding in order to eliminate molding stresses which have been detected using a polarized light inspection technique. Final selection of a case and component material is being deferred until further comparison of PPO and polysulfone is made.

Although polysulfone frames exhibit substantially less dimensional instability than PPO, frame warpage still presents a problem. Along with the use of flexible frame sealants, the use of more flexible frames is being investigated. Promising preliminary results using resilient frames molded in place around the electrode-separator cell pack have been obtained and further work will be done to explore this approach.

3.0 WORK PLANNED

During the next quarter, the 5 Ah multiple plate cells specified at the end of Task II will be made and evaluated. These cells will be subjected to the environmental tests, sterilization, cycle tests and other evaluation specified by the work statement.

Work will be done on frame materials and sealants to determine the best materials and technology for use in the cells which will be fabricated in Task III.

Additional work will be done on molding polysulfone and PPO in order to improve the overall quality of case, cover and other plastic components. Based upon the results obtained, the best materials will be selected.

The work devoted to selection of a top closure material for the cell pack will be completed and the best material will be selected.

Cycle tests on component test cells and multiple plate cells now in progress will be continued.

4.0 PERSONNEL

11,817 engineering hours have been expended on this project as of 24 July, 1966. The personnel who have worked on this project and the approximate percentage of their time devoted to this program are as follows:

Dr. C. Berger	No direct charge
F. C. Arrance	100%
A. Himy	50%
A. Rosa	100%
Q. McKenna	100%
H. Smith	100%
R. Sheridan	50%
M. Fajans	30%

REFERENCES

1. Technical Directive No. 1, April 19, 1966, D. G. Soltis, Project Officer, NASA Lewis Research Center Contract NAS 3-7639.

2. Berger, C; Arrance, F. C.; Taylor, A. D. and Himy, A. H.; Program to Develop an Inorganic Separator for a High Temperature Silver-Zinc Battery, Quarterly Progress Report SM-48461-Q2, NASA CR-54919, Astropower Laboratory, Douglas Aircraft Company, Inc., Newport Beach, California, March 1966.

3. Berger, C.; Arrance, F. C.; Taylor, A. D. and Himy, A. H.; Program to Develop an Inorganic Separator for a High Temperature Silver-Zinc Battery, Quarterly Progress Report SM-48461-Q3, NASA CR-54919, Astropower Laboratory, Douglas Aircraft Company, Inc., Newport Beach, California, April 1966.

APPENDIX A

TECHNICAL DIRECTIVE NO. 1 -- CONTRACT NAS. 3-7639

Section 7, Task II calls for the fabrication of eight (8) multiplate cells and subjecting these to a series of environmental and electrical tests. This evaluation follows component evaluation on three-plate test cells, Section 1-6, Task II.

Task III then calls for the fabrication and testing of two (2) lots of fifteen (15) cells each and the evaluation of these as described in the subject task.

In accordance with our discussions held at Astropower on March 3, 1966, the decision was made that before proceeding with Section 7, Task II, we should first construct five (5) multiplate cells to evaluate the proposed design effectiveness and preclude any obvious constructional errors. We would then proceed with the work called for in Section 7, Task II. The following tests shall be performed on each 5 ampere-hour cell to make this determination:

1. Phenolphthalein Leak Test
   (a) performed after each pressure test.
2. Pressure Test at 30 psig
   (a) dry and wet at 25°C
   (b) wet at 100°C
3. Capacity tests at 25°C and 100°C (two cycles)
   20 mA/cm^2 discharge to 1.0 volt, 4-5 mA/cm^2 charge
   10% overcharge
4. Heat soak at 135°C for three hours (discharged)
5. Internal Resistance Test

   At the completion of these tests, the cells shall be charged at c/20 and then given a short input pulse (5 - 10 seconds) at a rate of c in amperes. The cell voltages V_1, immediately prior to the pulse and V_2, 5 milliseconds after the pulse, are read on a suitable recording instrument. The internal resistance of the cell in ohms is then calculated.

$$R = \frac{V_2 - V_1}{I_c - I_{c/20}}$$

V_1 and V_2 are in volts; I_c and $I_{c/20}$ are in amperes.

6. Cell Short Test - loss of voltage on stand (two days)

The five cells used for this preliminary screening would therefore reduce the first lot of fifteen (15) cells to be constructed in Task III to ten (10) cells, with the second lot remaining at fifteen (15) cells.

Following these tests, the cells shall be evaluated by destructive examination.

Sincerely yours,

Daniel G. Soltis
Project Manager
Solar & Chemical Power Branch

APPENDIX B

TEST PROCEDURES FOR NASA CELLS IN ACCORDANCE WITH TD #1

1. Pressure test at 30 psig and phenophthalein leak test.

   --Wash cover and terminals thoroughly with water.

   --Dry

   --Apply paper wetted with phenophthalein solution over terminal seals and cover-to-case seal.

   (a) Dry test at 25°C:

   --Apply 30 psig pressure for 5 minutes and observe if phenophthalein reacts. Apply more solution if necessary.

   (b) Wet test at 25°C

   --Fill the cell with KOH.

   --Wash again and dry.

   --Wet again with reagent.

   --Apply 30 psig for 5 min at 25°C.

   --Observe reaction.

   (c) Wet test at 100°C

   --Place cell at 100°C for 2 hours.

   --Apply 30 psig for 5 minutes.

   --Remove and test again with phenophthalein.

2. Capacity Test:

   Run 3 cycles:

   Charge: All charges to be at R.T. at 350 mA to 2.05 V or 24 hrs, whichever occurs first.

   Discharge: 1 cycle at 25°C : 1.5 A to 1.0 V

   1 cycle at 25°C : 3 A to 1.0 V

1 cycle at 100 °C : Place cell in oven and start discharge: 3 A to 1.0 V

3. Internal Resistance Test:

--Charge the cell at I_1 = 0.250 A up to 1.92V minimum and let it run on this plateau for 10 minutes.

--Then apply a short pulse of I_2 = 5 A for about 5 to 10 seconds.

--Record voltage V_1 immediately prior to the pulse, and voltage V_2 during the course of the pulse.

--Calculate the internal resistance = $R = \frac{V_2 - V_1}{I_2 - I_1}$

--Repeat procedure 3 times for each cell.

4. Cell Short Test: (OCV test for 2 days at R.T.)

--Complete charge at 350 mA to 2.05 volts

--Leave cell on open circuit for 2 days at R. T.

--Monitor and record OCV every 2 hours during daytime with the same voltmeter.

5. Heat-soak test:

Discharge at 1.5 A to 1.0 V and drain the cell at 0.5 A to 1.0 V.
Then place the cell in an oven at 135 °C for 3 hours.

6. Recharge at 350 mA to 2.05 V at R. T.
Discharge at 1.5A to 1.0 V.